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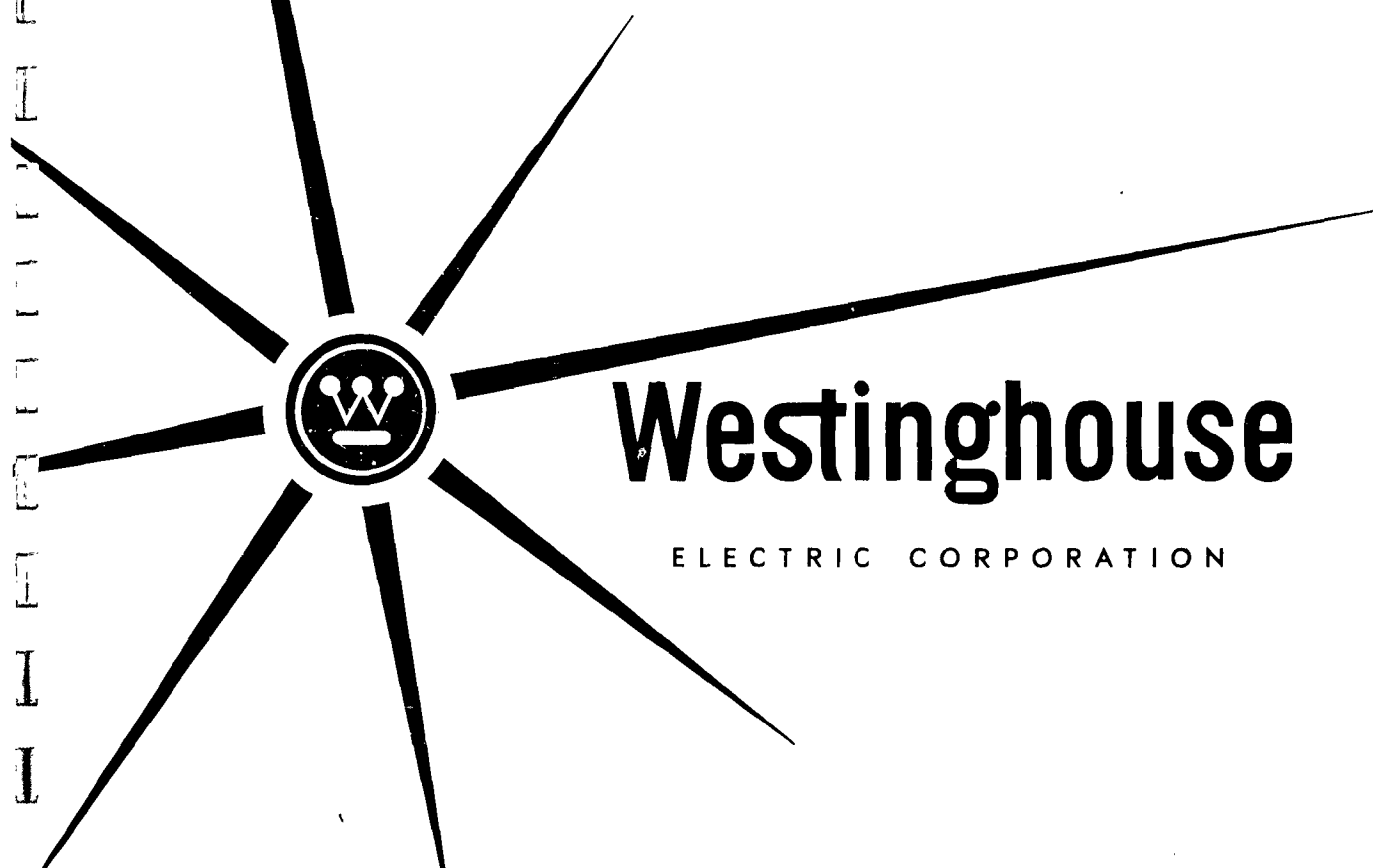
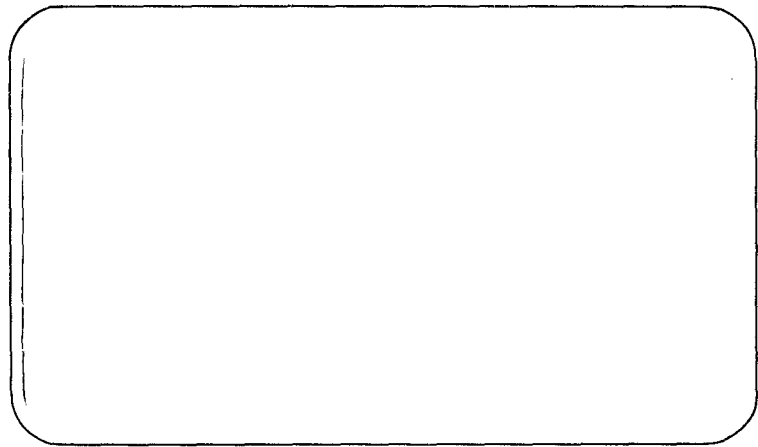
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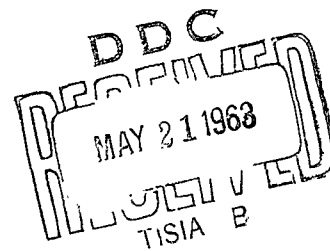
AEROSPACE ELECTRICAL DIVISION

ALKALI METAL RESISTANT WIRE

USAF CONTRACT AF33(657)10701

PROJECT NO. 8128

TASK NO. 8128-08



WESTINGHOUSE ELECTRIC CORPORATION

AEROSPACE ELECTRICAL DIVISION

LIMA, OHIO

DEVELOPMENT OF HIGH TEMPERATURE
ALKALI METAL RESISTANT INSULATED WIRE

1st Quarterly Progress Report
Contract AF33(657)10701, Task No. 8128-08

May 15, 1963

Westinghouse Electric Corporation
Aerospace Electrical Division
Lima, Ohio

NOTICE

The work covered by this report was accomplished under Air Force Contract AF33(657)10701, but this report is being published and distributed prior to Air Force review. The publication of this report, therefore, does not constitute approval by the Air Force of the findings and conclusions contained herein. It is published for the exchange and stimulation of ideas.

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FORWARD

This report is submitted by the Aerospace Electrical Division, Westinghouse Electric Corporation, Lima, Ohio, on Air Force Contract AF33(657)10701, Task No. 8128-08, "Development of High Temperature Alkali Metal Resistant Insulated Wire". The contract is administered by the Aeronautical Systems Division, Wright Patterson Air Force Base, Dayton, Ohio. Mr. Lester Schott is project engineer.

The work described in this report was carried out by personnel at the Research and Development Center, Department of Insulation and Chemical Technology, Westinghouse Electric Corporation, Pittsburgh, Pennsylvania.

ABSTRACT

This report covers the progress during the first quarter of Air Force Contract AF33(657)10701. The program effort is directed toward the development of an insulated electrical conductor resistant to saturated potassium (850 C) and mercury (538 C) vapors. Surveys of candidate insulators, conductors and coating methods are substantially completed. Exposure of a number of candidate insulators, uninsulated conductors, and metal-ceramic seals in potassium and mercury vapor were initiated to provide preliminary corrosion data. The mercury vapor exposure tests are completed. Evaluation of a number of coating methods were initiated and preliminary results from the work are promising. The difficulty encountered in finding a good method to effect a seal between the lead-in insulator on the electrical test exposure capsules prevented initiating electrical tests on the candidate insulating materials during the quarter.

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SECTION I

INTRODUCTION

Rotary power sources for advanced weapon systems based on nuclear reactors as energy sources utilize liquid metals as the working fluid to drive the turbines. Since the alternator used to supply the electrical energy is attached to the turbine shaft, its electrical insulation would be exposed to any of the metal vapor leaking through the seals. Present electrical insulation will probably be severely attacked when exposed to high temperature mercury vapor or alkali metal vapor such as potassium. In order to provide reliable electrical power, the present insulation must be protected from metal vapor by stator canning techniques.

Under this contract, a program was initiated to investigate insulation materials, electrical conductors, and coating methods needed in the development of a high current (4000 amps per square inch) round wire for advanced electromagnetic alternators that are exposed to mercury and alkali metal vapors. Saturated mercury vapor at 538 C was chosen to provide a realistic vapor pressure in test cells. Saturated potassium vapor at 850 C was chosen as a representative alkali metal vapor condition. The design objective life of the insulated conductor in the metal vapor environment is 10,000 hours. The resistance of the conductor at test temperature during its life is not to exceed 150% of the copper standard at 850 C. The room temperature tensile strength of the conductor is to be in excess of 30,000 psi. The initial purity of the potassium used in the exposure tests is 99.97%.

It is preferred that the conductor be insulated with a compatible high temperature insulation that is resistant to metal vapor attack, however, if this is not possible, then a potting compound compatible with the insulation and resistant to the metal vapor will be evaluated. The electrical strength from conductor to ground should have a design objective of 1200 volts. If the insulated conductor is potted, the electrical strength of the insulation should be at least 300 volts per mil.

The final evaluation of the insulated wire will be done in statorettes. The insulated conductor will be wound into statorettes to investigate winding techniques. While in the statorette, the insulation system will be subject to metal vapors, temperature, thermal shock, nuclear radiation, vibration, mechanical shock, humidity and acceleration.

SECTION II

SUMMARY AND PROGRAM MILESTONES

1. The survey of insulating materials was completed and 41 candidate materials which are commercially available were selected for further consideration.

2. The survey of electrical conductors was completed and three candidate conductors were selected for each metal vapor exposure in the preliminary corrosion tests.

3. Design of the corrosion test capsules was completed and the metal vapor exposures of the preliminary corrosion tests were initiated on a number of candidate insulating materials, uninsulated conductors and metal-ceramic seals. Mercury exposures at 538 C for up to 340 hours were completed and potassium exposures at 850 C are still in progress. The nickel plated copper conductors completely disintegrated in the mercury exposure tests.

4. The survey of coating methods is essentially completed and experimental work was initiated on three of the most promising methods. These methods are vapor deposition, plasma spraying and fusion bonding.

5. Design of the exposure capsule for electrical testing of insulating materials and insulated wire in the metal vapor environment was completed. Six methods of achieving the lead-in insulator seal to the capsule are under evaluation. •

SECTION III

EXPERIMENTAL WORK AND DISCUSSION

3.1 Materials

3.1.1 Insulation Materials - A list of 41 candidates has been prepared of materials that are good electrical insulators at temperatures above 1000 F. This data is presented in Table I. The data indicate considerable variation in T_e values for some materials. This is primarily due to variations in impurities content. A comparison, using any manufacturer's data available, will be made to prevent duplication and unnecessary testing. Single crystal and

high purity polycrystalline oxides are being obtained from various suppliers who are making these materials on a research or development scale.

A number of these materials have been obtained and are currently under test in the two environments. These initial exposures should shed some light on the possibility of forming conductive compounds on the insulation material surfaces particularly in the potassium vapor. Formation of such compounds would entirely rule out any imperfect or crazed insulating coatings. The insulation materials exposed to the mercury vapor at 538 C are listed in Table II. The materials under exposure to the potassium at 850 C are listed in Table III.

3.1.2 Electrical Conductors - The available information on electrical conductors suitable for extended service at 850 C and 538 C was reviewed. Even though copper may present possible grain growth problems at 850 C as reported by Anaconda⁽¹⁾ in their work on AF33(616)7473, we feel that the great limitation imposed on coating methods and materials by silver's low melting point (960 C) justifies continued consideration of copper cores for the potassium vapor environment. The grain growth effects only the ultimate tensile strength of the conductor and would only effect the electrical properties if the grains slip one on the other to cause a reduction of cross section of area on the wire at isolated points. The use of refractory metal cladding could prevent slipping at the grain boundaries. The larger diameter of the wire we will work with (#8 or #10 versus the #18 and #30 used by Anaconda) will provide a

finer initial grain size due to fewer annealing treatments. In addition to OFHC copper, cores of zirconium copper and dispersion hardened (Thoria, Beryllia) copper will be considered as means of preventing grain growth.

Duplicate specimens of nickel plated copper (10% Ni), nickel clad copper (28% Ni) and stainless steel clad copper (28% type 410) were exposed to mercury vapor at 538 C for 340 hours. Weight and appearance changes are listed in Table I. The nickel plated copper conductors completely disintegrated during the test and portions of the copper core in the nickel clad conductor disappeared probably due to pinholes in the cladding. Micrographs are being made of the wires that remained intact. Details of the test are in the section titled Corrosion Studies.

Identical samples of the wire are under exposure to potassium vapor at 850 C. Scheduled for exposure in potassium are tantalum clad copper, Inconel clad copper with a tantalum barrier layer, tantalum clad silver, nickel clad silver and Inconel clad silver (12%-88% by vol). The Ni and Inconel clad silver wires should be available from Anaconda. The other commercially unavailable wires will be made by the rod and tube method.

Lengths of .050" and .100" OFHC copper have been obtained for use in preparing iron and chromium plated copper. Discussions were held with vendors such as Tubotron, Inc., about sheathing insulated conductors with a resistant metal if a wire insulation or potting compound with adequate resistance to the metal vapor attack cannot be found. Tubotron currently produces an aluminum sheathed

polyethylene insulated copper wire. The sheathing is done by high frequency welding of the aluminum by a technique similar to the Thermotool process of New Rochelle Tool Co.

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3.2 Coating Methods

The survey of coating methods is essentially complete and a number of the techniques uncovered for coating wire are being investigated because they have been used successfully to apply pure oxide coating materials. These methods are vapor deposition, thermal decomposition of vapors and gases, anodization, flame and plasma spraying, fusion and solution ceramics.

Actual laboratory coating trials were initiated on the following three methods during the first quarter.

3.2.1 Plasma Coatings - Several pieces of Inconel cut to a 1/16 x 3/8 x 1-1/2 inch shape to fit into tubes used in the environmental tests have been plasma sprayed with alumina. The alumina used was the highest purity grade supplied by the Metco Co. for use in their equipment. The coatings achieved were not as dense as previous samples and had an electric strength of less than 200 vpm.

Previously plasma sprayed coatings of magnesium oxide are available and samples of these coatings along with the alumina coatings will be tested in alkali metal vapor environments.

3.2.2 Fusion Coatings - The sintering of very finely divided oxides is possible at temperatures much lower than conventionally needed with coarser forms of the oxide. The addition of very small

amounts of silica have been shown to materially reduce this lower temperature even further. Although silica is generally considered detrimental to resistance to potassium vapor, Cowan⁽⁴⁾ of the Los Alamos Scientific Lab reported that up to 1% silica in alumina was tolerable in cesium vapor at 2200 F. Since coating compositions of finely divided oxides could be continuously fused onto conductors using a tube furnace or RF heating, this approach looks very promising.

Compositions using alumina and magnesia with and without silica have been prepared for use with Inconel clad copper conductors.

3.2.3 Vapor Deposition - Thermal decomposition of "B" trichloroborazole in the vapor phase with deposition of boron nitride⁽²⁾ is under investigation. A two zone furnace has been constructed and used in preliminary trials at forming the BN on platinum. If the two zone furnace proves unsatisfactory, RF heating will be tried. Results of initial coating trials are encouraging and the work will be continued during the next quarter.

During the next quarter, evaluation of the three coating methods listed above will continue and work will be initiated to evaluate vacuum deposition, solution ceramics and anodization.

3.3 Corrosion Tests

During the first quarter, the corrosion test capsule was designed, facilities for handling, filling and sealing the capsules were installed, and preliminary corrosion tests were initiated.

3.3.1 Capsule Design and Loading Technique - Figure 1 contains a detailed drawing of the corrosion capsule along with a flow diagram illustrating the technique used to clean, fill and seal the capsule. Capsule design and sealing techniques have proven satisfactory by the successful completion of preliminary exposure tests in mercury for 340 hours without leakage. In addition, exposure of an evacuated 316 stainless Swagelok cap used in this design at a temperature of 850 C for 250 hours has not resulted in any leakage. This result indicates that the sealing technique can also be used for potassium vapor corrosion tests.

Early plans to seal the capsule by crimping were not successful. It was found that crimping the tube ends did not provide a vacuum tight seal, thus preventing the removal of the capsule from the dry box for the final cutting and welding operation necessary with this technique.

The present design uses a 1/2" diameter 304 seamless stainless steel tube of .025" wall thickness. One end is sealed by a crimping/welding operation while the other end, after capsule filling, is sealed in a vacuum with the use of a 1/2" Swagelok cap (Cat. #810-C-316).

In addition, a large dry box capable of evacuation to 1×10^{-3} torr has been procured and installed. It is fed with dry argon from a NaK bubbler operating at 600 F and has been equipped with an electric wrench capable of tightening Swagelok fittings of 1" maximum diameter remotely under a high vacuum environment.

3.3.2 Cover Gas Purification Facilities - The argon cover gas is purified in a bubbler using NaK 78 at 600 F. The resulting O₂, N₂ and H₂O content is below the sensitivity of our mass spectrometer (< 50 ppm). The potassium used in the preliminary tests is obtained from MSA Research Corp. and has an oxide content of < 100 ppm. Oxide content of the potassium will be determined when necessary by the mercury amalgamation technique described by Champiex, et al⁽³⁾. The mercury metal as obtained from the supplier is about 5 ppm contaminate.

3.3.3 Aging Facilities - Two Lindburgh furnaces have been installed in the liquid metal lab for use in corrosion studies. This brings to three the number available for such work. All furnaces are equipped with exhaust ducts to remove any vapors generated in the event of a faulty capsule leaking during the 1000 hour exposure test.

3.3.4 Corrosion Tests - A number of preliminary corrosion tests have been made on various wire and insulator materials in a mercury vapor environment at 538 C. The results of these tests are shown in Table II. Nickel plated, nickel clad and stainless clad copper core wire samples and various oxides of aluminum and boron phosphate were exposed to the Hg vapor for 340 hours and 260 hours respectively. Ends of the wire samples were welded closed to prevent amalgamation of the copper core. Although the ceramic samples were discolored by the exposure, their resistance to mercury vapor based on weight loss appears to be good at 538 C. Nickel plated copper core wire was severely attacked by the vapor, primarily be-

cause the plate was apparently not continuous. The greatest penetration occurred in that location where the wire was bent in order to place it in the capsule. Of the three wires tested, 410 stainless steel clad wire showed the least attack although its surface was blackened.

Preliminary potassium exposure tests were initiated later in the quarter. A summary of the materials presently being tested at 850 C in potassium vapor is given in Table III. These tests will run for the 1000 hour cycle.

3.4 Electrical Tests in Metal Vapor Environments

3.4.1 Electrical Test Capsule Design - The capsules will be fabricated from 0.50 inch OD tantalum tubing with 0.020 inch wall. Disks fabricated from candidate insulation will be fitted into one end of the tube and a seal will be effected using one of six possible techniques. The remainder of the capsule sealing will be done using Swagelok fittings as in the corrosion exposure capsules. In the initial tests, the lead-in disk will serve as the test specimen. An electrode can be sealed in a hole centered in the disk by means of a glass seal. The capsule wall will act as the second electrode. The surface resistivity of the annular space will be measured as a function of time and applied voltage. These tests will determine any gross attack i.e., extreme corrosive attack or formation of conducting films such as potassium aluminate. Finding a completely satisfactory material for the disk is relatively simple compared to the conductor insulation problem since the simple shape would allow use of single crystals, if necessary.

Six methods are under consideration for effecting the seal between the lead-in insulator and the capsule. These are:

- a. alumina-silicate glass seal
- b. electron beam welding
- c. heat shrink fit seal
- d. high temperature pressure bonding
- e. brazing to a metallized ceramic
- f. titanium diffusion bonding

The alumina-silicate glass initially selected for the capsule seal does not appear as attractive as originally thought. This seal material has been used in a cesium vapor environment at elevated temperatures. The device containing the seal had hot spot temperatures of 1000 C. The actual seal area was at a temperature considerably less than 1000 C. In addition, some very preliminary tests conducted to determine the resistance of this sealing glass to 850 C potassium vapor has yielded some questionable results. Further testing is planned, however, the other sealing methods listed above are being considered in the event that the glass proves unsatisfactory.

The use of tantalum as the capsule material to match the glass co-efficient of expansion would require either doing the aging of the capsules in vacuum or heavily plating the capsules with chromium to prevent degradation of the tantalum. Tantalum capsules aged in air at 850 C completely disintegrated in 24 hours.

Except for the aluminum-silicate glass, the methods for effecting the seal are listed in order of probability of success. Nickel, Kovar or stainless steel would be the material of construction for the capsules with the remaining seal methods.

Metal-ceramic seal combinations exposed in mercury and potassium vapor in the preliminary corrosion tests are listed in Tables II and III. Attempts at fabrication of actual capsules with sealed lead-in insulators have been started.

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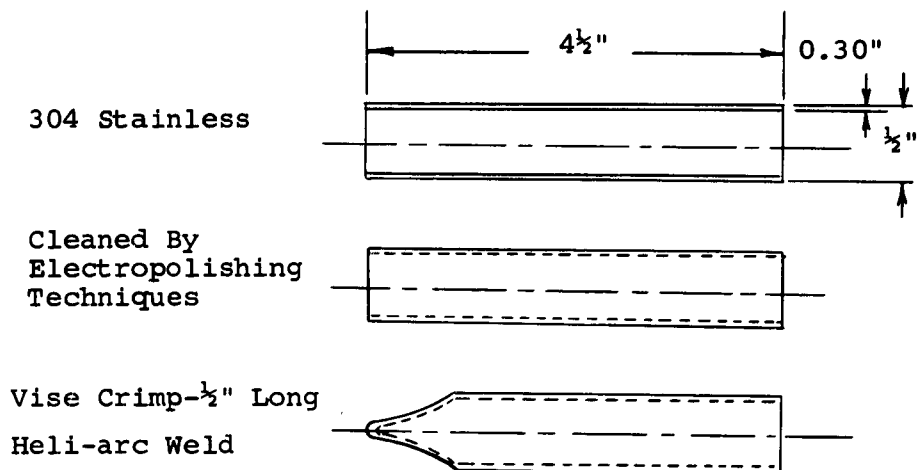
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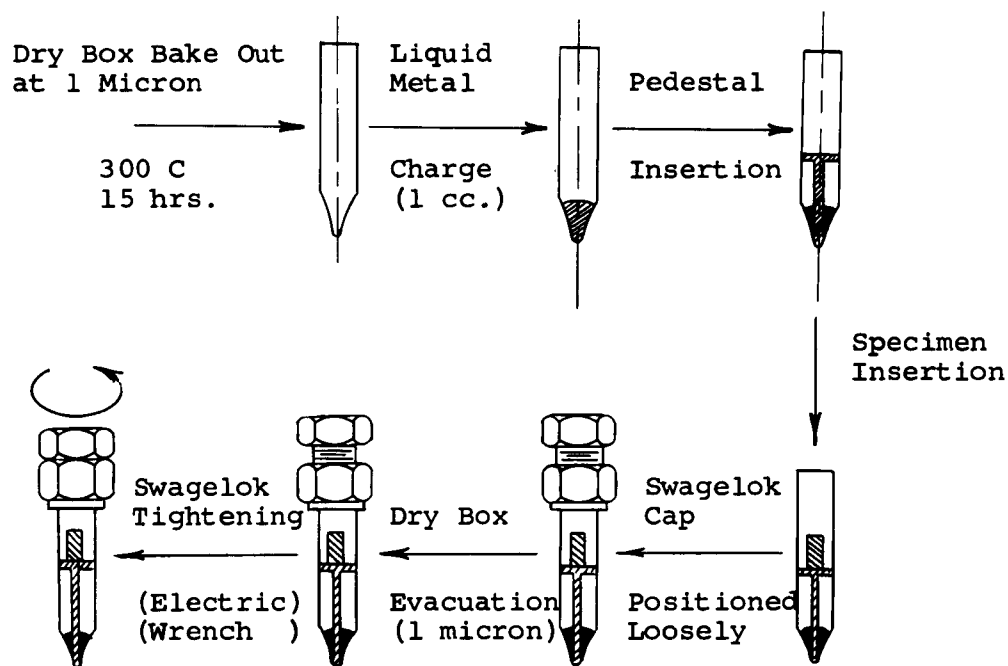
SECTION IV

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- (2) R. J. Patterson et al., Thin Films of Boron Nitride, presented at the 123rd Electrochemical Society Meeting, Pittsburgh, Pa., April 1963.
- (3) Champiex et al., Journal of Nuclear Materials, Vol. 1, pp. 113-119, 1959.
- (4) Robert E. Cowan, Stephan D. Stoddard, Ceramic Materials for Nuclear Thermionic Converters, presented at the 65th Annual Meeting of the American Ceramic Society, May, 1963.



Note: Weld is leak checked with helium after heating to cherry red with oxy-acetylene torch.



Note: Pedestal, Sample & Swagelok baked out at 300 C - 1 micron 15 hrs.

Figure 1 - Cleaning and sealing technique for corrosion test capsules

TABLE I

INSULATING MATERIALS WITH T_e^* VALUES ABOVE 1000 F

MATERIAL	TRADE NAME OR DESIGNATION	SUPPLIER	T_e VALUE IN °F
1. Porcelain	BV600	Gen. Ceramics	1112
2. Zirconium Oxide	Alsimag 508	Amer. Lava	1130
3. Aluminum Silicate	Lava Grade A	" "	1148
4. Zirconium Oxide	Alsimag 550	" "	1157
5. Steatite	#13809	Frenchtown Porcelain	1170
6. Porcelain	BV582	Gen. Ceramics	1202
7. Alumina	Alsimag 491	Amer. Lava	1238
8. Zircon	#3569	Frenchtown Porcelain	1251
9. Zircon	Alsimag 504	Amer. Lava	1292
10. Cordierite	#5301	Frenchtown Porcelain	1369
11. Steatite	Alsimag 196	Amer. Lava	1382
12. Cordierite	Alsimag 202	" "	1436
13. Alumina	Alumicox #4462	Frenchtown Porcelain	1472
14. Alumina	ADL210	Gen. Ceramics	1472
15. Magnesium Silicate	Lava 1136	Amer. Lava	1490
16. Steatite	Alsimag 228	" "	1508
17. Alumina	Alsimag 393	" "	1535
18. Steatite	Alsimag 197	" "	1544
19. Alumina	Alsimag 491	" "	1544
20. Steatite	BN3942	Gen. Ceramics	1562
21. Zircon	M-81-A	" "	1562
22. Alumina	AB-2	Coore	1562
23. Zircon	Alsimag 475	Amer. Lava	1598
24. Alumina	AL-100	Coore	1679
25. Alumina	Alite 212	U. S. Stoneware	1787
26. Thoria	Thorox	Nat. Beryllia	1830
27. Magnesium Silicate	BN3055	Gen. Ceramics	1832
28. Forsterite	BN3054	" "	1832
29. Alumina	AL-300	Western Gold & Plati- num	1832
30. Forsterite	Alsimag 243	Amer. Lava	1832
31. Magnesium Silicate	Alsimag 222	" "	1832
32. Alumina	1009	Western Gold & Plati- num	2012
33. Alumina	AL-200	Coore	2138
34. Magnesia	Alsimag 555	Amer. Lava	2192
35. Alumina	Alox	Nat. Beryllia	2250
36. Beryllia	Berlox	" "	2400
37. Strontium Zirconate		Zirconium Corp.	1870
38. Alumina + Yttria		" "	--
39. Magnesia (Single Crystal)		Muscle Shoals Electro Chem. Co.	--
40. Boron Nitride		Carborundum Co.	--
41. Alumina (Single Crystal)		Linde	--

* T_e is the temperature at which a material has an insulation resistance value of one megohm-cm.

TABLE II

WIRE AND CERAMIC EXPOSURE TESTS - MERCURY VAPOR @ 538 C

Sample No.	Sample Ident.	Mercury Charge	Test Time-hrs	Weight Before (gms)	Info After (gms)	Weight Change, gms	Surface Area	Comments
WH-1	Ni plated Cu lcc (10% Ni)	"	340	.8084	No Sample	Remaining	--	Sample completely disintegrated
WH-2	"	"	"	.8143	"	"	--	(Same as WH-1)
WH-3	Ni clad Cu (28% Ni)	"	"	1.2136	1.0514	-.1622	--	Portions of copper core missing; holes in Ni clad.
WH-4	"	"	"	1.2162	1.1192	-.0970	--	(Same as WH-3)
WH-5	St. St. clad Cu (28%-410)	"	"	1.2245	1.1410	-.0835	--	Stainless steel blackened.
WH-6	"	"	"	1.2172	1.1890	-.0282	--	" "
CH-1	Al203-AD 99	"	260	.8712	.8719	+.0007	.484	Black surface
CH-2	Lucalox	"	"	.5360	.5363	+.0003	.434	" "
CH-3	BPO4	"	"	1.2328	1.2334	+.0006	.922	" "
CH-4	Saureisen #8	"	"	.6645	.6636	-.0009	.496	Grey surface
CH-5	Lucalox/Ta	"	"	.2371	.2366	-.0004	--	Ceramic separated from Ta
CH-6	Al203/Ta	"	"	.3741	.3747	+.0006	--	No visible change in seal. Al203 darkened.

TABLE III
WIRE AND CERAMIC EXPOSURE - POTASSIUM VAPOR @ 850 C

Sample No.	Sample Ident.	Potassium Charge	Test Time-hrs	Weight Before-gms	Information	Surface Area-in ²
WK-1	Ni plated Cu (10%)	1 cc		.8211		--
WK-3	Ni clad Cu (28%)	"		1.2562		--
WK-5	S. S. clad Cu (28%-410)	"		1.2929		--
CK-1	AL203-AD 99	"		1.1397		.626
CK-2	Lucalox	"		.3493		.299
CK-3	BPO ₄	"		.4746		.457
CK-4	Saureisen #8	"		.6035		.422
CK-7	Al203 (Coran)	"		3.1059		1.01
CK-8	Tho ₂	"		.8262		.320
CK-9	ZrO-CaO	"		1.8735		tube
CK-10	MgO (hot pressed)	"		4.9116		1.392